

ULTRASONIC CHARACTERIZATION OF CERAMIC-CERAMIC COMPOSITES

J. B. Walter, L. A. Lott, and K. L. Telschow

Idaho National Engineering Laboratory
EG&G Idaho, Inc.
Idaho Falls, ID 83415

INTRODUCTION

Ceramic-reinforced ceramic matrix composites are being developed for high temperature, high strength applications such as heat engine components, heat exchangers, and recuperators.[1] The particular material considered here is a SiC-fiber-reinforced SiC matrix composite formed by chemical vapor infiltration (CVI). The initial focus was on porosity, which has been linked to the material strength.[1] Ultrasonic attenuation and velocity were investigated since they were expected to be affected by the porosity in the material.[2-4]

The objectives of this study were to determine the capability of conventional methods to measure the ultrasonic properties of the materials, and develop improved methods where necessary; to determine the correlations between ultrasonic properties and material properties of interest; and to demonstrate the ability of the ultrasonic technique to nondestructively characterize ceramic composite components.

EXPERIMENTAL PROCEDURES

Samples

A set of SiC/SiC samples designed to have a wide range of porosity levels was made specifically for this program. The samples were in the form of plates measuring, nominally, 100 x 38 x 3.3 mm fabricated from preforms built up from multiple layers of SiC cloth.^a The SiC matrix was added by a chemical vapor infiltration (CVI) process^b which results in samples with high porosity because, as matrix continues to deposit on the fibers in the preform, further infiltration of the gaseous reactants can be inhibited. The bulk porosity varied between 26 and 39 vol% for the samples examined.

Experimental Setups

The materials studied here highly attenuated the ultrasonic waves. Through-transmission techniques were developed because no back surface

a. Nicalon, Nippon Carbon Co., Tokyo, Japan.

b. Refractory Composites, Inc., Whittier, California.

echoes were evident for conventional pulse/echo techniques. Both conventional piezoelectric and laser generation of ultrasonic pulses were used to study the attenuation and velocity. The advantage of the laser technique is that greater ultrasonic energy is coupled into the sample, permitting investigation of a smaller portion of the sample with a given measurement.

Ultrasonic measurements were made in pulsed, through-transmission. Figure 1 shows the standard arrangement using two broadband, 2.25 MHz, 12.7 mm diameter, piezoelectric transducers. An ultrasonic pulse was generated by one of the transducers, and received by the other using a standard pulser/receiver. The voltage response of the receiving transducer (A-scan) was recorded. The attenuation and velocity were determined from the A-scan as described below.

Measurements were also made with the laser generation setup shown in Figure 2. A Nd:YAG laser heated the sample surface with a 10 ns pulse. For most of the area scans, the laser was operated at 10 mJ/pulse, which was distributed over a 6 mm spot on a dry sample surface. An ultrasonic pulse was produced at the surface via thermoelastic conversion.[5] (Other modes of ultrasonic generation become important when the energy density is considerably greater, or when the sample surface is wetted.) The attraction of this technique is production of the ultrasonic pulse at the surface of the sample, thereby avoiding potentially large losses due to reflection at the first surface. Larger ultrasonic energy densities were obtained with this setup than with the piezoelectric setup; for example, a 92 mJ laser pulse provided 20 dB more gain than a broadband 5 MHz, 6 mm diameter, piezoelectric transducer. Consequently, smaller receiving transducers could be used to obtain more localized ultrasonic measurements. The spot size was selected to (a) match the receiving transducer, (b) reduce the energy density in the laser pulse, and (c) average out effects from surface roughness and reinforcement distribution.

The A-scan in Figure 3 illustrates the complexity of the received ultrasonic signals. This scan, recorded with the laser system, is typical of signals obtained with both methods of ultrasound generation. A low frequency pulse (approximately 0.8 MHz) arrives first and is followed by smaller voltage fluctuations which persist for tens of microseconds. The nature of these fluctuations has not been determined, though they are probably associated with multiple scattering in the material rather than back surface echoes. The apparent peaks in these fluctuations shift drastically as the sample is moved across the laser beam, suggesting interference and phase cancellation.

Ultrasonic Measurements

Ultrasonic attenuation was measured with the piezoelectric system by considering only the ultrasonic signal within a window of 1.5 μ s beginning with the initial arrival of the pulse. This method includes the first arriving main pulse but ignores the trailing ultrasonic signals. The response with the sample present was compared to the response with the sample removed to obtain the insertion loss. Correction was made for losses at the sample surfaces by calculating a reflection coefficient from the bulk density and ultrasonic velocity through the plate. Conventional diffraction corrections were very small compared to the high attenuation losses in the samples and were ignored. Measurements were made for each plate at an array of points distributed over a 20x40 mm area centered on the sample. Each A scan was Fourier transformed to the frequency domain, and the results averaged to obtain a representative value for the plate.

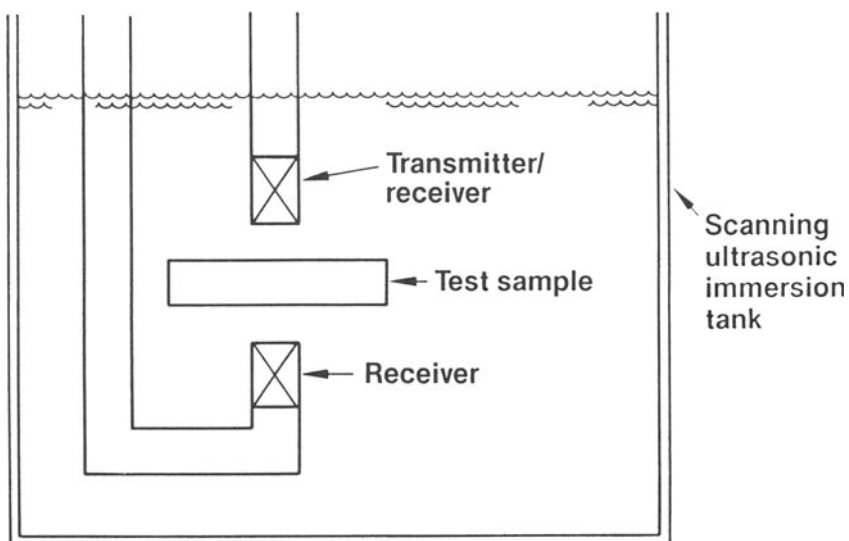


Figure 1. Through-transmission measurement system using two piezoelectric transducers coupled to the sample by the water bath.

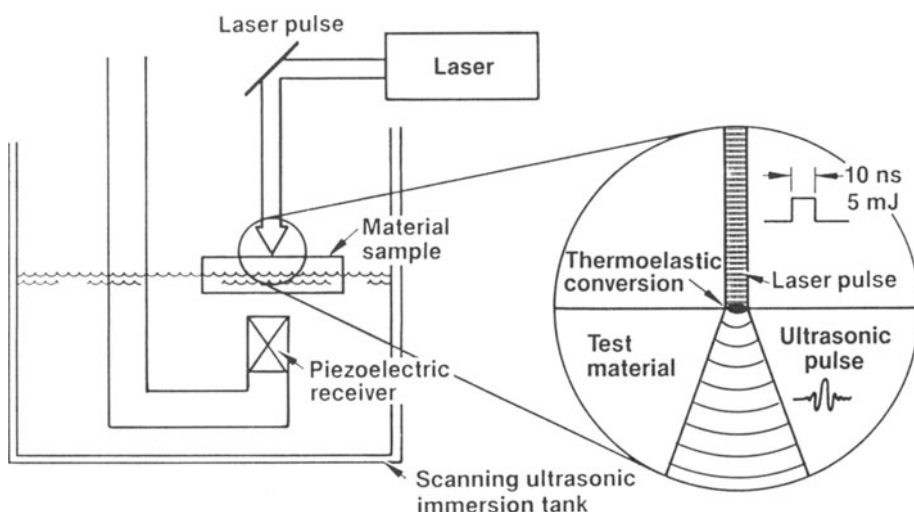


Figure 2. Through-transmission measurement system using a pulsed laser to generate an acoustic pulse at the surface of the sample by thermoelastic conversion. The transmitted acoustic pulse is detected by a conventional piezoelectric transducer, coupled to the sample by a water bath.

Variations in the distribution of porosity within a plate were studied by mapping the energy in the transmitted ultrasonic pulse. The transmitted energy is a direct measure of attenuation in a sample, but requires significantly less signal processing than does calculation of the actual attenuation coefficient. It was thus a very efficient means of rapidly scanning a large number of samples. The ultrasonic energy was calculated from the A scan for a 0.5 μs window starting with the arrival of the pulse. This energy depends on the transmission characteristics of the sample, as well as on the frequency characteristics of the initial ultrasonic pulse and of the receiving transducer. However, the latter two quantities are constant for a particular setup, so a map of transmitted energy of a sample will show only variations in sample attenuation. It is a nonunique condensation of the frequency dependent insertion loss to a single number which can be readily presented in a grey scale or pseudocolor map of a plate.

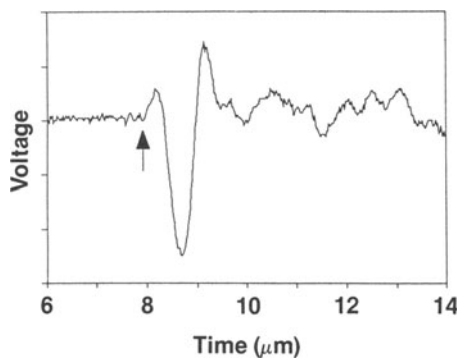


Figure 3. Example of the recorded voltage output from the receiving transducer in the thermoelastic setup. The arrow marks the arrival of the pulse at about 8 μs . The implied velocity is 4.5 km/s, corresponding to 1.5 μs between multiple echoes within the sample.

The ultrasonic velocity normal to the plate was determined from the time of arrival of the ultrasonic pulse at the receiving transducer. For the piezoelectric system, the method of Markham[6-8] was used. For the laser system, a sample with known velocity was used to determine the time of travel in the water couplant. In either case it was necessary to extract an accurate time-of-arrival from the A scan. The center of the ultrasonic pulse is not an acceptable reference point because the samples act as low pass filters, and, consequently, the received pulse contains only the low frequency components of the initial pulse which makes it much broader. The beginning of the pulse, the only feature in the A-scan which does not depend on the attenuation of the sample, can be determined by fitting a straight line to the leading edge of the pulse and extrapolating

it back to the point of departure from the baseline. The estimated accuracy was 10 ns, the digitizing period of the transient recorder, which translates to an accuracy in the velocity of a few percent.

RESULTS

The highly attenuating nature of the SiC/SiC samples produced by CVI is illustrated in Figure 4. The sample with 26% porosity shows an attenuation varying from 3 to 11 dB/mm, and the sample with 39% porosity has about 10 dB/mm greater attenuation. Thus each back surface echo, for a 3 mm thick plate with 26% porosity, would be down in amplitude from the previous echo by a factor of 8 (18 dB) at 800 kHz, and by a factor of 2000 (66 dB) at 2.3 MHz. Some of this "lost" energy is showing up as the persistent signal, obscuring the diminutive back surface echoes.

The correlations between the porosity and the attenuation and velocity measurements are presented in Figures 5 and 6, respectively. Each error bar is the standard deviation of the measurements for the corresponding plate, and is indicative of the degree of uniformity. The lines through the data are the result of unweighted least squares analyses, and give changes per percent porosity of 0.5 dB/mm for attenuation (at 1.6 MHz) and 0.16 km/s for velocity.

Ultrasonic maps of a sample reveal nonuniform distributions of porosity. A sample with particularly large variations in porosity is featured in Figure 7. The X-ray radiograph in Figure 7a, which was calibrated by a series of penetrameters, reveals that the porosity ranges from as low as 31% in the dark region to as high as 43% in the light region. The ultrasonic maps show the same general pattern as does the radiograph - a darker band curving down from the upper left, across the middle, to the upper right. The lower porosity region transmits more energy, and has higher velocity. The range of velocities in Figure 7c, about 3 to 5 km/s, is consistent with the velocity measurements in Figure 6.

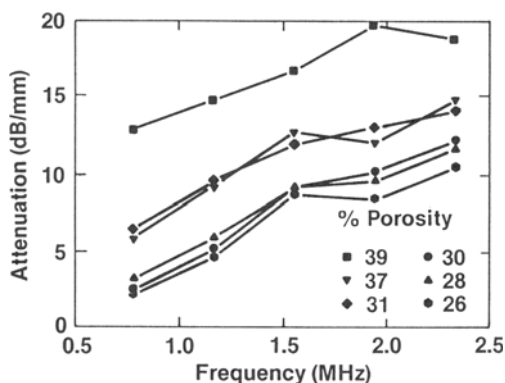


Figure 4. Attenuation measured for six SiC-SiC samples with porosity content ranging from 26 to 39 volume percent. The attenuation increases by about 5 dB/MHz.

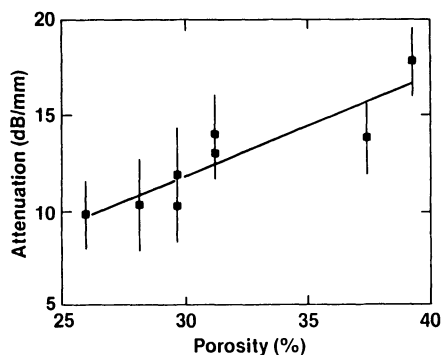


Figure 5. Attenuation at 1.6 MHz as a function of sample porosity. The attenuation increases at about 0.5 dB/% porosity. The error bars are the standard deviation values measured at different points on the sample. Two sets of measurements are shown for two of the samples.

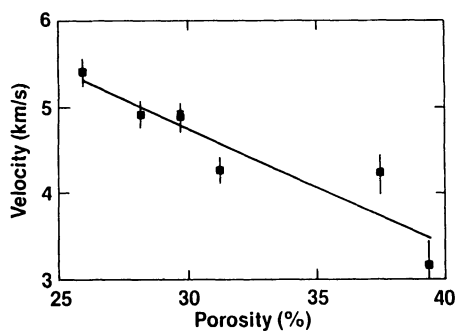
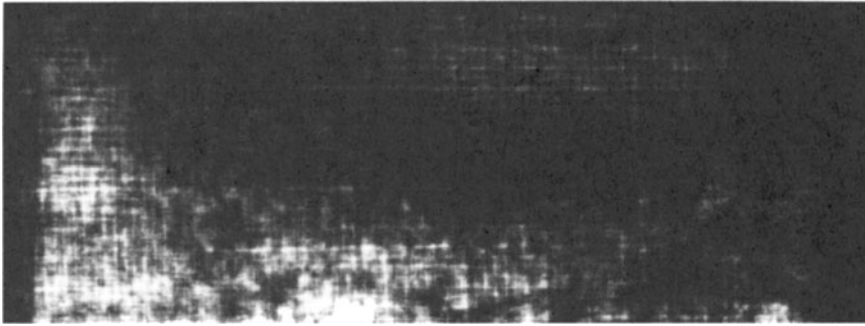


Figure 6. Acoustic velocity measured with the conventional pulsed system. Velocity is seen to depend strongly on the sample porosity. The error bars are the standard deviation of measurements at different points on the sample. Two sets of measurements are shown for one sample.



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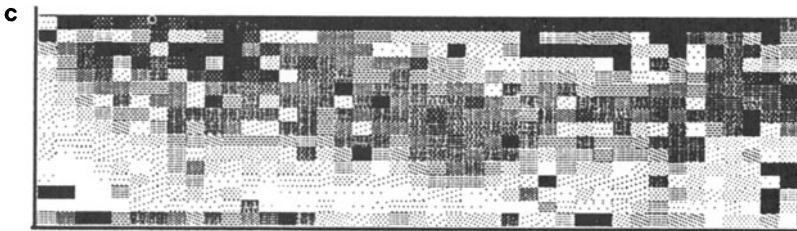
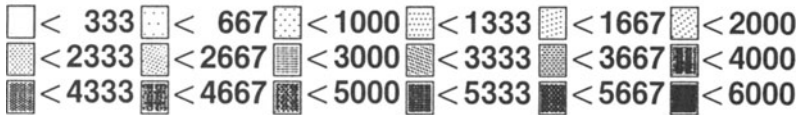
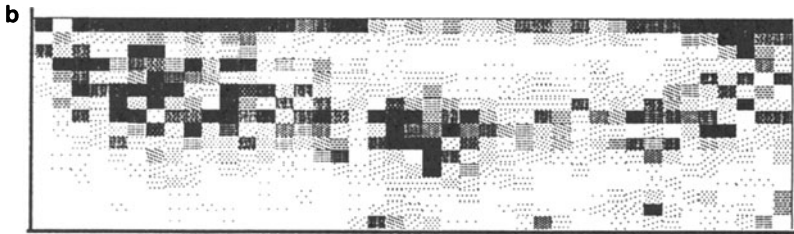


Figure 7. SiC-SiC sample with porosity variations from 31% to 43%. a) Positive print of X-ray radiograph, and maps of b) the transmitted ultrasonic energy (darker = higher energy), and c) the ultrasonic propagation velocity (darker = higher velocity).

CONCLUSIONS

Knowledge of basic ultrasonic properties of advanced ceramic/ceramic composite materials is required to develop ultrasonic NDE techniques for these materials. Because of the nature of ceramic composites, ultrasonic measurements are difficult to make, requiring special measuring techniques to be developed. Experimental measuring techniques have been developed to measure ultrasonic wave propagation velocity and attenuation.

Ultrasonic properties were measured in through-transmission at low frequencies (<5 MHz), using both conventional piezoelectric generation and laser generation of the ultrasonic pulse. Better spatial resolution was afforded with the laser technique, because the higher ultrasonic energy densities in the sample permitted the use of a smaller receiving transducer.

The ultrasonic attenuation and velocity show strong correlation to the material porosity. However, the degree of scatter evident in the data indicates that other factors also have important influences on the propagation of ultrasonic energy through these SiC/SiC plates.

ACKNOWLEDGMENT

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